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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
YUUKI TAUCHI, ET AL. : EXAMINER: MORILLO, J.C.  
SERIAL NO: 10/633,550 :  
FILED: AUGUST 5, 2003 : GROUP ART UNIT: 1793  
FOR: AG BASE ALLOY THIN FILM AND :  
SPUTTERING TARGET FOR FORMING  
AG BASE ALLOY THIN FILM

DECLARATION UNDER 37 C.F.R. §1.132

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Yuuki TAUCHI who deposes and states that:

1. I have a masters of engineering degree, which was conferred upon me in 2001 by Tsukuba University located in Ibaraki, Japan.
2. I have been employed by Kobe Steel, Ltd. since 2001 and been seconded to Kobelco Research Institute, Inc., which is a wholly-owned subsidiary company of Kobe Steel, Ltd, for 2 years , and I have a total of 7 years of work and research experience in the field of thin film material (for optical disk).
3. I understand the English language or, at least, the contents of this Declaration were made clear to me prior to executing the same.
4. The following experiments were carried out by me or under my direct supervision and control.

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5. The following is description of the experiments performed herein and the importance of the same.

6. To establish the criticality of the lower limit of Bi content in the present invention of 0.005 at%, we performed the following experiment. Using alloy targets, each thin film of Ag-Bi alloys (sample Nos. 1 to 4), with a film thickness of 100 nm (as a reflective film), or 15 nm (as a semi-transmissive reflective film) was deposited on a polycarbonate substrate (diameter: 120 mm, thickness: 0.6 mm) with a DC magnetron sputtering process using a sputtering equipment, HSM-552 from SHIMADZU EMIT Co.,LTD. Then, each composition of these Ag base alloy thin films were examined by an ICP (inductively coupled plasma) mass spectroscopy.

Then, using each Ag base alloy thin film deposited, the durability of a reflective film (film thickness 100 nm) or a semi-transmissive reflective film (15 nm) were examined.

#### *6.1 Evaluation of thermal stability*

The reflectance of each thin film with a thickness of each of the 100 nm-thick thin films deposited in the foregoing manner was measured by means of spectrophotometer, V-570 from JASCO Inc.

Then each of the samples was subjected to a high-temperature high-humidity test (temperature 80 °C - humidity 90 % RH – retention time 48 hours). After the test, the reflectance was measured again. For the evaluation, the one showing absolute values of the changes in reflectance before and after the high-temperature high-humidity test of 5 % or less (wavelength 405 nm) and 1 % or less (wavelength 650 nm) has been judged as having high durability. The results are shown in Table 1.

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Table 1

Sample No.	Composition	Change in reflectance before and after high temperature high humidity test [%]		High durability
		wavelength 405nm	wavelength 650nm	
1	Ag-0.004at%Bi	-5.49	-1.42	×
2	Ag-0.010at%Bi	-3.32	-0.79	○
3	Ag-0.020at%Bi	-1.42	-0.08	○
4	Ag-0.055at%Bi	-1.12	0.09	○

○: GOOD, ×: NO GOOD

As is apparent from Table 1, any of the Ag base alloy thin films of the sample Nos. 2 to 4 satisfying the defined requirements of the present invention has high durability. In contrast, for the thin film of the sample No. 1, which contains less than the lower limit of Bi than defined in the present invention, it is not possible to obtain a prescribed high durability.

## 6.2 Evaluation of chemical stability

Each of the 15 nm-thick thin films deposited in the foregoing manner was subjected to a salt immersion test (salt water concentration: 0.05 mol/l for NaCl, salt water temperature: 20 °C, immersion time: 5 minutes). The changes in appearance of the thin film after the test were visually observed. For the evaluation, the one of which the changes in appearance such as discoloration and peeling were not observed have been judged as having high durability. The results are shown in Table 2.

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Table 2

Sample No.	Composition	Change in appearance after salt solution immersion test	High durability
1	Ag-0.004at%Bi	Yes	×
2	Ag-0.010at%Bi	No	○
3	Ag-0.020at%Bi	No	○
4	Ag-0.055at%Bi	No	○

○: GOOD, ×: NO GOOD

As apparent from Table 2, any of the Ag base alloy thin films of the sample Nos. 2 to 4 satisfying the defined requirements of the present invention has high durability. In contrast, for the thin film of the sample No. 1, which contains less than the lower limit of Bi than defined in the present invention, it is not possible to obtain a prescribed high durability.

7. In addition to the demonstration of criticality of 0.005 at% Bi as the lower limit of Bi, in the Declaration under 37 C.F.R. §1.132 filed on February 7, 2008, I presented data evidencing the criticality of 0.4 at% Bi as the maximum Bi desired. The purpose of the data presented below is to show that the effect of Bi fully commensurate in scope with the instant claims, e.g. even at the content of the claimed minimum Bi. Further, the effect of the above-mentioned property could be obtained even when the Nd is contained.

The upper limit of Bi content in silver alloy reflective film for optical disk is set at 0.4 atomic percent desirably because modulation of recorded signal decreases and error signal increases in terms of decreasing thermal conductivity of reflective film by more than 0.4% addition of Bi.

The characteristics of Ag-Bi alloy are higher thermal conductivity/higher reflectivity/higher durability, and high thermal conductivity which is required for high speed recording optical disc is not obtained by addition of Bi more than 0.4 atomic percent as

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described in the specification. Changes in thermal conductivity of film by adding Bi is described in the tables presented in the examples.

On recording optical disc for example, DVD-R, recording mark is formed by heat arisen by irradiating laser beam into recording layer (dye). High thermal conductivity is required for reflective layer adjacent to recording layer for high speed recording because it is necessary to diffuse heat rapidly for forming recording mark in shorter time. In case of using low thermal conductivity material for reflective layer, a mark would be influenced heat arisen by forming former mark and appropriate recording mark wouldn't be formed. As a result, PI error which is a symbol value for error signal becomes worse, as shown in the Declaration under 37 C.F.R. §1.132 filed on February 7, 2008.

As the reflective layer material, Ag alloy containing 0.005at% Bi or further addition of Nd, has a relatively high thermal conductivity as shown in the example of the specification, appropriate recording mark would be formed. As a result, PI error which is a symbol value for error signal would be well.

8. The following Example was performed:

#### *8-1. Preparation*

A dye layer for DVD+R as recording layer was coated by spin coating process on 0.6 mm thickness polycarbonate substrate having groove as specified in DVD+R specification and annealed for drying. Subsequently, a Ag, Ag-Bi or Ag-Bi-Nd reflective layer was deposited by sputtering process. DVD bonding machine (NEC engineering co., Ltd.) was used for dye coating. Unaxis Cube-star was used for sputtering process. Sputtering power was set at 2kW, and sputtering time was set at about 4 seconds as film thickness should be 140nm. DVD+R disks having 1.2 mm thickness were obtained with bonding 0.6 mm thickness polycarbonate substrate by DVD bonding machine with UV resin.

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## 8-2 Evaluation

### 8-2-1 Bi content in film

Samples for Bi and Nd content analysis were prepared by sputtering with same condition for making reflective layer in DVD+R disks above on polycarbonate substrate. Bi and Nd contents were analyzed by ICP (inductively coupled plasma) spectroscopy and ICP mass method.

### 8-2-2 Electric signal property

BenQ DW1640 was used for recording on DVD+R disks. Each disk was recorded by 4X. PI error was evaluated by Nero CD-DVD-speed.

9. The following results were obtained:

Table 3 shows PI of manufactured disks.

Bi content (at%)	Nd content(at%)	PIE [count]	Electric property
0	0	172	GOOD
0.02	0	48	GOOD
0.049	0	66	GOOD
0.05	0.2	111	GOOD
0.07	0.7	57	GOOD

PI error (PI sum 8) should be 280 or less was established by Standard so that data of PI error of 280 or less was evaluated as GOOD. PI error has been maintained under 280 when the Bi content is near lower limit, 0.005at%. PI error also has been maintained under 280 when the Nd is further contained. Accordingly, it is proper that Bi contents of film should be 0.005at% to 0.4 at% when the film will be used as reflective layer of DVD+R Disk. Further, it is proper that Nd is further contained.

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10. I declare further that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

11. Further Declarant saith not

田内 裕基

Name: Yuuki TAUCHI  
Kobe Steel, Ltd.

10/27/2008

Date